



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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| (51) International Patent Classification ⁵ : A23G 3/30, A23L 1/058, 1/22 A23L 1/226 | A1 | (11) International Publication Number: WO 92/02145 (43) International Publication Date: 20 February 1992 (20.02.92) |
| (21) International Application Number: PCT/US91/05348 (22) International Filing Date: 29 July 1991 (29.07.91) (30) Priority data: 565,521 9 August 1990 (09.08.90) US (71) Applicant: WARNER-LAMBERT COMPANY [US/US]; 201 Tabor Road, Morris Plains, NJ 07950 (US). (72) Inventors: CHERUKURI, Subraman, Rao ; 10 Jean Drive, Towaco, NY 07082 (US). MANSUKHANI, Gul ; 97 Petrus Avenue, Staten Island, NY 10312 (US). (74) Agents: SCOLA, Daniel, A., Jr. et al.; Warner-Lambert Company, 201 Tabor Road, Morris Plains, NJ 07950 (US). | | (81) Designated States: AT (European patent), AU, BE (European patent), CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FI, FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, KR, LU (European patent), NL (European patent), NO, SE (European patent). Published <i>With international search report.</i> |
| (54) Title: FLAVOR DELIVERY SYSTEM WITH IMPROVED RELEASE AND METHOD OF PREPARATION (57) Abstract <p>A flavor delivery system is disclosed which offers the combination of improved delayed flavor release with improved extension and reduces bitterness, and when incorporated into chewing gums, provides desired softness to the gum to facilitate the reduction in softening additives that conventionally add moisture thereto. The present delivery system comprises a composite of a flavor, a resin and a polyalkylene wax, preferably in a solution mixture with each other, the flavor being present in an amount of from about 1.0 % to about 90 % by weight of the final delivery system, the resin being present in an amount of from about 0.5 % to about 80 % by weight of the final delivery system, and the polyalkylene wax being present in an amount of from about 0.5 % to about 90 % by weight of the final delivery system. Optionally, an emulsifier may be added in an amount of up to 25 % by weight. In a further embodiment, the delivery system of the present invention may include up to 25 % by weight of a sweetener such as acesulfame-K. The present delivery system finds use in comestibles such as chewing gum compositions and other food products, pharmaceuticals, and scratch-and-sniff and aroma packaging products. The delivery system may be incorporated as a liquid or may be prepared in particulate form.</p> | | |

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⁺ It is not yet known for which States of the former Soviet Union any designation of the Soviet Union has effect.

**FLAVOR DELIVERY SYSTEM WITH IMPROVED
RELEASE AND METHOD OF PREPARATION**

RELATED APPLICATIONS

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The present Application is a Continuation-in-Part of Co-pending Application Serial No. 361,529, filed June 5, 1989, in the names of the inventors herein. Applicants claim the benefit of this application under 35 U.S.C.

10 Section 120.

BACKGROUND OF THE INVENTION

This invention relates to a new delivery system for
15 flavors and to the process for its preparation, which has use in a variety of products including comestibles such as chewing gum compositions, confections, pharmaceuticals, food products such as beverages and some baked goods, dentifrice compositions and denture
20 adhesives and in packaging applications. More particularly, this invention relates to a process for preparing a flavor delivery system which facilitates the delivery of higher levels and intensity of flavor without the harshness or off notes that are experienced at higher
25 flavor concentrations.

Flavor delivery systems are well known in the art which may be divided into various classes based upon their physical states, namely, liquids, emulsions, pastes or
30 solids. Not only are these states different but the potential uses for each state also differ as do their method of manufacture.

Over the years considerable effort has been directed
35 toward the preparation of flavoring materials. Specifically, flavor materials have been sought that provide greater flavor intensity coupled with sustained flavor release for long periods of time.

An outgrowth of this activity has been the observation that when free flavor oil is added to gum base, only about 5% to 40% of the initial flavor oil is released from the gum upon chewing. It appears that as much as
5 80% of the remaining flavor oil becomes irreversibly bound to the gum base and cannot be chewed out.

To overcome this difficulty with flavor oils in particular, various attempts have been made to
10 encapsulate the flavor oils or use dried ingredients to inhibit the gum base binding action. In addition, considerable effort has been directed to the development of delayed release flavoring agents that will delay
15 release of the flavoring agent while permitting uniform release of the flavor over an extended period of time. The obvious benefit of immediate/delayed release is the ability to provide uniform flavor sensation during the entire consumption period which has not been previously attainable by the direct incorporation of conventional
20 seasonings and flavor oils into chewing gum formulations.

Spray drying is one of the most widely used techniques to encapsulating or fixing a flavor. In this procedure a flavor oil is usually blended with a film forming agent
25 dispersed in water and then emulsified to form a stable emulsion. Emulsification is necessary because flavor oils are insoluble in the water needed to dissolve the film forming agent. Obtaining an emulsion of low viscosity, at high solids content, is critical to
30 producing an acceptable product. Stability at higher temperatures is also critical to avoid volatilization of the flavor oil. Maintaining low inlet temperatures (i.e. around 155°C) as well as minimizing the residence time in the dryer are beneficial if the flavor is chemically
35 unstable. The air drying conditions depend on the emulsion and product characteristics such as particle

size of the product required. Modifications to the use of heat to remove the water from the emulsion have also been attempted. Some of these include the use of a dehydrating solvent as the drying medium rather than hot
5 air. Freeze drying has also been contemplated.

Flavor fixation has also been obtained by the extrusion method wherein the flavor oil is coextruded with a water soluble sugar or sugar mixture, dried and ground for use.
10 These products find application in dry mixes for instant release of the flavor on contact with water. Such products generally contain 10 to 15% by weight of flavor oil.

15 An extensive discussion of the prior art with respect to the extension of flavor is found in U.S. Patent No. 4,590,075 to Wei et al. The patentees discuss the preparation of encapsulated flavors which are added together with a quantity of a non-confined hydrophobic
20 flavor oil to a suspension agent before formulation in a chewing gum (U.S. Patent No. 3,920,849 to Marmo et al.); the encapsulation of flavor in high molecular weight material such as polyvinyl acetate (U.S. Patent No. 3,826,847 to Ogawa et al.); the preparation of a flavor
25 composite by crosslinking the flavor with a water-insoluble hydrophilic polymer such as hydroxyethyl acrylate (U.S. Patent No. 3,761,286); the preparation of microencapsulated flavor particles and their dispersion in a slurry on the surface of a chewing gum (U.S. Patent
30 No. 3,962,463). Further, U.S. Patent No. 4,695,463 to Yang et al. is noted and discloses a flavor delivery system that relies on the immobilization of the flavor within a crosslinked hydrocolloid multivalent alginate or carageenenate matrix.

35

Further, U.S. Patent No. 4,597,970 to Sharma et al.

prepares a delivery system for actives such as sweeteners and including flavors which utilizes a hydrophobic matrix comprising edible fatty acids or waxes, lecithin and a glyceride component. This system is urged to provide a protective barrier and controlled release characteristics to the active so contained. The combination of materials in the encapsulating matrix provides wetting capabilities with respect to non-uniform core materials such as aspartame, while providing improved heat stability and thereby, extended high temperature processing capability. These attributes were frequently achieved at a cost of delay in release of the active.

U.S. Patent No. 3,867,556 to Darragh et al. also encapsulated volatile flavors in a fat or wax material. The patentees had found that the initial or base encapsulation displayed excessive instability to heat, and as their product was intended primarily for incorporation into baked goods, they applied a second coating of a water-soluble material such as gum arabic, which would provide high temperature stability while conferring rapid disintegration on contact with moisture. The Darragh et al. product likewise exhibited delayed flavor release.

Further, U.S. Patent No. 4,515,769 to Merritt et al. disclosed the encapsulation of flavor material in a partially hydrophilic matrix comprising gelatin, a natural gum or albumin or a plasticizer. This combined encapsulant was placed in an emulsion with the flavoring agent and thereafter dried to a solid matrix which was then ground to a base powder. The base powder was then coated with a water-insoluble material which purported to prevent flavor loss and delay flavor release in contact with moisture. Merritt et al. sought only to achieve a burst of flavor when the physical mastication of their

flavor composite ruptured the outer encapsulating material.

U.S. Patent No. 4,087,557 to Bakal et al. relates to the
5 incorporation of an artificial sweetener directly into
the ester gum component of a chewing gum base, to achieve
a delay and corresponding extension in sweetener release
(column 2, lines 31-42). The patent contemplates the
incorporation of certain flavors, specifically, food
10 acids such as tartaric acid, succinic acid, etc. directly
into the ester gum component, however, indicates that
conventional flavors such as flavor oils are added
separately after the gum base constituents including the
artificial sweetener containing-ester gum have been mixed
15 with each other and cooled. The flavor component is
incorporated into chewing gums prepared in accordance
with the patent in the conventional fashion, i.e., by
direct incorporation of artificial flavor and/or by the
incorporation of flavor coated with a hydrophilic
20 material such as gum arabic (see Example 6). Bakal et
al. accordingly offers no solutions to the problems of
flavor delivery and extension of concern to the art.

Other approaches to the preparation of flavor composites,
25 particularly with flavor oils, are noted. Thus, U.S.
Patent No. 3,041,180 to Swisher discloses a process for
extruding a flavor oil with glycerine and corn syrup
solids to form an encapsulated extruded flavor oil. The
solid was dried and yielded a particulate solid. U.S.
30 Patent No. 4,610,890 to Miller et al. discloses another
process for preparing an extruded flavor oil with sugar,
a starch hydrolysate and an emulsifier.

U.S. Patent Nos. 4,448,789 and 4,569,851 to Yang
35 discloses a novel flavoring agent -- hydrophilic
polymer -- by blending the flavor with the polymer and

blending while heating to prepare a homogenous product. Once coupled and ground, the product can be used as a flavoring for chewing gum and confectionery products.

5 U.S. Patent No. 4,271,202 to Giel relates to a spray-drying process for forming solid flavoring material capable of including high percentages of flavoring oil per total particulate unit of weight. Because of the high oil content possible, such spray-dried products
10 found wide use in a number of beverages and other foods. However, it was also found that spray-dried flavors prepared by this and similar known methods typically exhibit a relatively limited shelf life. In addition, the high temperatures necessarily involved during spray-
15 drying processes have been found to impair the flavor and aroma of various heat-sensitive oil flavors, such as those in citrus fruit. Furthermore, solids formed by spray-drying commonly exhibit hygroscopic characteristics making them difficult to handle and store.

20 In addition to these techniques, U.S. Patent No. 4,452,821 to Gergely is directed to a confectionery product, especially a chewing gum, that purports to offer prolonged, extended delivery of flavoring aroma and/or
25 active pharmaceutical ingredient. This is accomplished by providing the flavoring, aroma or active pharmaceutical ingredient in a solid solution or mixture within a wax containing functional groups, said wax forming a homogeneous mixture of solid solution. The wax
30 does not contain any functional groups and is substantially immiscible with the flavoring, aroma or active pharmaceutical ingredient.

The approaches to flavor modification discussed above all
35 fall short of providing a flavor delivery system which achieves the advantages of flavor intensity and fullness

inherent in flavor oils in combination with improved release and extension characteristics. In most instances, the gum base and, in particular, the elastomer component sequesters the flavor and thereby prevents its release during the chew. Moreover, the plasticizers and softeners conventionally employed in gum formulations tend to increase moisture pickup and corresponding flavor migration and loss, so that the flavor that might be available for release on chewing is further diminished in content and resulting extent of release and sensation.

In earlier filed co-pending Application Serial No. 329,742, it was proposed to prepare a flavor delivery system comprising a liquid flavor, preferably a flavor oil, and a resin component selected from wood rosins and ester gums and mixtures. The delivery system offers a combination of improved release and intensity, however when higher concentrations of flavor are sought to be added, the resulting delivery system exhibits some of the harshness or off notes that are characteristically present in such circumstances.

A need, therefore, exists for the development of a flavor delivery system which remedies the aforementioned problems by preventing or at least minimizing the demonstration of harshness by offering a combination of delayed release and desirable masking to the flavor component, while at the same time promoting improved flavor intensity and longevity, and increased amount of flavor release from the chewing gum base in combination with desirable organoleptic properties and reduced moisture pickup.

SUMMARY OF THE INVENTION

The present flavor delivery system comprising a composite flavor having improved flavor retention, softening and

delayed flavor release characteristics in combination with reduced bitterness when incorporated into chewing gums and other products, is prepared with a liquid flavor, preferably a flavor oil, a resin component
5 selected from wood rosins and ester gums and mixtures, and a polyalkylene wax. The flavor component is preferably a flavor oil and is present in an amount by weight of the total composite of from about 1.0% to about 90%, the resin component comprises from about 0.5% to
10 about 80% and the polyalkylene wax is preferably a polyethylene wax and is present in an amount of from about 0.5% to about 90%.

More particularly, the flavor component is present in an
15 amount from about 35% to about 65% by weight, the resin component is present in an amount of about 20% to about 55% by weight, and the polyalkylene wax is present in an amount of from about 5.0% to about 50% by weight. A particularly preferred formulation comprises from about
20 30% to 45% by weight of flavor, from about 35% to about 55% by weight of resin, and from about 2.5% to about 15% by weight of the polyalkylene wax. The polyalkylene wax may be a polyethylene wax and may have a molecular weight ranging from about 500 to about 10,000, and mixtures of
25 waxes having differing molecular weights are contemplated.

The present composite may be prepared by heating the resin component to its melting point followed by adding
30 the polyalkylene wax component under agitation until a homogeneous mixture and solution results. Thereafter, the flavor component may be added to the resin-wax solution mixture, and this second mixture agitated until homogeneity. Optionally, an emulsifier may be initially
35 added to the resin-wax mixture and the resulting composite will comprise a composite emulsion. The

present composite flavor is generally liquid or semi-liquid in state, depending upon the amount of resin present, and the melting points of the resin and flavor components.

5

The primary advantage of the flavor delivery system of the present invention flows from the taste masking effect that the polyalkylene wax component exerts on the flavor component, and the improvements in the delay and
10 extension of flavor release characteristics. This permits the preparation of the composite with higher concentrations of flavor, which in turn results in increased flavor perception and longevity, and improved stability and delivery even after high temperature
15 processing.

A further advantage of the present flavor delivery system resides in its generally liquid state. Specifically, the final flavor-resin-wax composite is a liquid and may be
20 directly incorporated into chewing gum formulations in the liquid state, thereby reducing the amount of moisture added during gum formulation and consequently, reducing the moisture content of the final gum product. The present flavor composite acts as a softener to the
25 elastomer component of the gum base and thereby reduces the amount of conventional softeners such as glycerin, corn syrup, sorbo liquid and gum arabic solution that must be added, so that the resulting gum formulation is less likely to exhibit undesirable moisture pickup. The
30 foregoing combination permits the formulation of low moisture gum formulations.

Moreover, both the resin and the wax components act as both a carrier and binder in that they tie up the flavor
35 and mask its aroma even at the higher temperatures at which the present composite is prepared, and at which

chewing gum compositions receiving the flavor composite are processed. An advantage, therefore, of this temperature stability is that flavor loss during product formulation employing the present flavor composite is substantially reduced.

The present composite finds utility in a variety of food and confectionery products as an additive thereto, and in a further embodiment, may be formulated with a sweetener in an amount that may range up to about 25% by weight thereof to offer a combined sensation of flavor and sweetness. For example and as illustrated herein, the present composite was formulated with 10% by weight of acesulfame-K and was found to provide an excellent presentation vehicle for the sweetener. The present delivery system provides the same protection to both actives and thereby enhances the longevity and intensity of both flavor and sweetness release that are experienced.

The composite may, accordingly, be incorporated into chewing gums and particularly into low calorie, low moisture formulations, as well as a flavorant in pharmaceutical preparations and as a part of tablet coatings, and in such novel products as "scratch-and-sniff" and aroma packaging.

The flavor delivery system of the present invention may be used alone or in combination with conventional flavors including spray dried flavors and the like. Also, although the present flavor delivery system is generally prepared and received as a liquid or semi-liquid, it can be prepared in solid form by such techniques as spray drying and the like.

In a further embodiment, the present invention includes a

chewing gum comprising a gum base, sweeteners, fillers, and other additives and the present flavor delivery system. Both sugar containing and sugarless gums are contemplated, including gums with high gum base-low carbohydrate content.

The flavor delivery system is prepared by heating the resin component to its melting point which, in most instances, ranges from about 85°C to about 90°C, after which the wax component is added and the resulting mass is heated with mixing to a temperature of from 100° to 110°C until homogeneous. Heating is then stopped and the resulting melt is cooled to a temperature of less than 85°C to minimize flashing off of the subsequently added flavor component. Thereafter, the resin is permitted to slowly cool while the flavor component or oil is added with agitation. Upon the completion of the addition of the flavor component, the resulting blend is continually stirred until it cools to approximately room temperature. The resulting blend may then be recovered and will be found to be generally liquid or semi-liquid, or clear or amber in color. Any additions of emulsifier take place prior to the addition of the flavor component so that the emulsifier and resin are mixed and melted together. Any additions of artificial sweeteners take place either simultaneously or sequentially after the addition of the flavor to the resin melt, with the remainder of the processing remaining essentially the same.

Accordingly, it is a principal object of the present invention to provide a flavor delivery system that offers delayed flavor release characteristics and reduced offnotes and bitterness.

It is a further object of the present invention to provide a flavor delivery system as aforesaid which

provides greater flavor capacity, intensity and longevity in a component which provides desired plasticity to chewing gums into which it is incorporated.

- 5 It is a still further object of the present invention to provide a chewing gum composition or other comestible product having contained therein the flavor delivery system of the present invention.
- 10 It is a still further object of the present invention to prepare a flavor delivery system having included therein an artificial sweetener for the improved delivery of both flavor and sweetness.
- 15 Other objects and advantages will become apparent to those skilled in the art from a consideration of the ensuing description which proceeds with reference to the following illustrative drawings.

20

BRIEF DESCRIPTION OF THE DRAWINGS

- Figure 1 is a graph comparing the flavor intensity and longevity of a hydrous, peppermint-flavored sugared gum composition containing the inventive delivery system,
- 25 with a control gum composition containing a combination of conventional spray dried and liquid peppermint flavor.

- Figure 2 is a graph presenting the results of comparative sensory evaluation testing of anhydrous sugarless chewing
- 30 gums containing the inventive flavor delivery system prepared with peppermint flavor and a control gum containing conventional spray dried and liquid peppermint flavor.

35

DETAILED DESCRIPTION

In accordance with the present invention, a flavor delivery system is disclosed which offers improved flavor protection and a combination of delayed and extended release characteristics and reduced bitterness, in conjunction with unexpected softening properties when incorporated into chewing gum compositions. The flavor delivery system accordingly comprises a non-aqueous solution mixture of:

- (a) a flavor component in an amount of from about 0.1% to about 90% by weight of the final delivery system;
 - (b) a resin component, said resin component comprising a rosin, in an amount of from about 0.5% to about 80% by weight of the final delivery system; and
 - (c) a polyalkylene wax in an amount of from about 0.1% to about 90% by weight of the final delivery system;
- wherein said resin component and said polyalkylene wax component protect, hold and mask the aroma of said flavor component even at high temperatures, whereby said flavor delivery system may be formulated with higher concentrations of said flavor component and exhibits said reduced bitterness and said combined delayed and extended flavor release.

More particularly, the flavor delivery system of the present invention comprises the flavor component in an amount of from about 35% to about 65% by weight of the final flavor delivery system, the resin component present in an amount of from about 20% to about 55% by weight thereof, and the polyalkylene wax which may be a polyethylene wax having a molecular weight ranging from about 500 to about 10,000, and which may be present in an amount of about 0.5% to about 50% by weight thereof.

In a particularly preferred embodiment, the flavor delivery system of the present invention comprises from

about 30% to about 45% by weight of the flavor component, from about 35% to about 55% by weight of the resin component and from about 2.5% to about 15% by weight of the polyalkylene wax component.

- 5
- Optionally, an emulsifier may be included, and in a preferred embodiment, the emulsifier may be present in an amount ranging up to about 25% by weight, preferably from about 10% to about 20% by weight, and most preferably
- 10 from about 5% to about 10% by weight. In the instance where emulsifiers are employed, suitable emulsifiers may include mono-, di and triglyceride esters of fatty acids, polyglycerol esters and the like. More particularly, the emulsifier may be selected from the group consisting of
- 15 lecithin, stearates, ester derivatives of stearates, palmitates, ester derivatives of palmitates, oleates, ester derivatives of oleates, glycerides, sucrose polyesters, polyglycerol esters and mixtures thereof.
- 20 Suitable polyalkylene waxes include the polyethylene waxes manufactured by Petrolite Specialty Polymers Group, Tulsa, Oklahoma and known as the Polywax® Polyethylenes. The waxes generally useful in the present delivery system are crystalline aliphatic hydrocarbons having molecular
- 25 weights that range from 500 to 10,000. A particular wax is known as Polywax® AC-6, which has a molecular weight of 600. Naturally, other polyalkylene waxes are contemplated, such as polypropylene waxes, and polyethylene glycol waxes, and the invention is not
- 30 limited to the specific waxes described hereinabove.

Though not wishing to be bound to a particular theory of operation, it is believed that the present invention is predicated on the discovery that an interaction exists

35 between flavors and particularly flavor oils and resins such as wood rosins and rosin esters and the polyalkylene

wax component, such that the resins and the wax protect and mask the flavor component, and thereby reduce its bitterness and offnotes, while delaying and extending flavor release. Accordingly, it is further theorized
5 that an increase in the amount of resin in solution with a flavor such as a characteristically volatile flavor oil, shields the aroma of the flavor oil and decreases its potent odor. Moreover, this protective effect is greatly enhanced when the polyalkylene wax is present, so
10 that the resulting system further reduces flavor harshness, and correspondingly permits higher flavor contents and increased availability and delivery of the flavor.

15 Useful flavoring agents may be chosen from synthetic flavoring liquids and/or liquids derived from plants, leaves, flowers, fruits, etc., and combinations thereof. Preferably, the flavor component is selected from spearmint oil, cinnamon oil, oil of wintergreen
20 (methylsalicylate) and peppermint oils. Also useful are artificial, natural or synthetic fruit flavors such as citrus oil including lemon, orange, grape, lime and grapefruit and fruit essences including apple, strawberry, cherry, pineapple and so forth.

25 The amount of flavoring agent employed is normally a matter of preference subject to such factors as flavor type, base type and strength desired. In general, amounts of about 0.5% to about 10.0% by weight of the
30 final chewing gum composition are usable with amounts of about 0.5% to about 2.5% being preferred and about 0.7% to about 2.0% being most preferred.

The term "resins" as used herein refers to resins such as
35 wood rosins, rosin esters and ester gums. Suitable resins accordingly may be selected from pentaerythritol

ester of partially hydrogenated wood rosin,
pentaerythritol ester of wood rosin, glycerol ester of
wood rosin, glycerol ester of partially dimerized rosin,
glycerol ester of polymerized rosin, glycerol ester of
5 tall oil rosin, glycerol ester of wood rosin and
partially hydrogenated wood/gum rosin and partially
hydrogenated methyl ester of rosin, such as polymers of
alpha-pinene or beta-pinene; terpene resins including
polyterpene and mixtures thereof.

10

The above indicated resin components useful in accordance
with the present invention are selected because of their
compatible temperature characteristics. Thus, the
glycerol ester of partially hydrogenated wood or gum
15 rosin, manufactured by Hercules Chemical and identified
as Resin Ester #5, has a softening point ranging from
about 79°-88°C. Similarly, the glycerol ester of tall
oil rosin has a softening point of about 80°-88°C; the
glycerol ester of wood rosin has a softening point of
20 about 88°C-96°C; the deodorized glycerol ester of wood
rosin has a softening point of about 88°-96°C; and the
methyl ester of partially hydrogenated wood rosin is
liquid at room temperature. Certain of the polyterpenes
comprising the terpene hydrocarbon resins have softening
25 points that vary with the type of resin and generally,
however, range from about 100°-125°C. Similarly, the
pentaerythritol ester of partially hydrogenated wood
rosin has a softening point of from about 102°-110°C; the
pentaerythritol ester of wood rosin has a softening point
30 ranging from about 109°-116°C; the glycerol ester of
polymerized rosin has a softening point ranging from
about 80°-126°C; and the glycerol ester of partially
dimerized rosin has a comparable softening point.

35 The present invention includes both sugar containing and
sugarless chewing gums comprising a gum base, one or more

sweeteners and the present flavor delivery system. In the instance where the gum formulations are being prepared, a variety of softening agents may be utilized for their intended purposes. Such softening agents
5 suitable for gum formulations include lecithin, glycerin, acetylated monoglyceride, triacetin, hydrogenated vegetable oils, mineral oils, polyglycerol esters of fatty acids, and mixtures thereof. These softening agents are utilized within conventional amounts for such
10 ingredients.

In accordance with a further embodiment, the present invention includes a chewing gum composition offering improved flavor intensity and longevity, comprising a gum
15 base, a sweetener and a flavor delivery system, said flavor delivery system offering improved flavor protection and a combination of delayed and extended release characteristics in conjunction with reduced bitterness, said flavor delivery system comprising a non-
20 aqueous solution mixture of:

(a) a flavor component in an amount of from about 1.0% to about 90% by weight of the final delivery system;

(b) a resin component, said resin component comprising a rosin, in an amount of from about 0.5% to
25 about 80% by weight of the final delivery system; and

(c) a wax component comprising a polyalkylene wax, in an amount from about 0.5% to about 90%;

wherein said resin component and said polyalkylene wax component protect, hold and mask the aroma of said
30 flavor component even at high temperatures, whereby said flavor delivery system may be formulated with higher concentrations of said flavor component and exhibits said reduced bitterness and said combined delayed and extended flavor release.

35

With regard to chewing gum formulations in which the

novel delivery system is employed, the amount of gum base employed will vary greatly depending on various factors such as the type of base used, consistency desired and other components used to make the final product. In

5 general, amounts of about 5% to about 85% by weight of the final chewing gum composition are acceptable for use in chewing gum composition with preferred amounts of about 15% to about 70% by weight. The gum base may be any water-insoluble gum base well known in the art.

10 Illustrative examples of suitable polymers in gum bases include both natural and synthetic elastomers and rubbers. For example, those polymers which are suitable in gum bases include, without limitation, substances of vegetable origin such as chicle, jelutong, gutta percha

15 and crown gum. Synthetic elastomers such as butadiene-styrene copolymers, isobutylene-isoprene copolymers, polyethylene, polyisobutylene and polyvinylacetate and mixtures thereof are particularly useful.

20 The gum base composition may contain elastomer solvents to aid in softening the rubber component. Such elastomer solvents may comprise methyl, glycerol or pentaerythritol esters of rosins or modified rosins, such as hydrogenated, dimerized or polymerized rosins or mixtures

25 thereof. Examples of elastomer solvents suitable for use herein include the pentaerythritol ester of partially hydrogenated wood rosin, pentaerythritol ester of wood rosin, glycerol ester of wood rosin, glycerol ester of partially dimerized rosin, glycerol ester of polymerized

30 rosin, glycerol ester of tall oil rosin, glycerol ester of wood rosin and partially hydrogenated wood rosin and partially hydrogenated methyl ester of rosin, such as polymers of alpha-pinene or beta-pinene; terpene resins including polyterpene and mixtures thereof. The solvent

35 may be employed in an amount ranging from about 10% to about 75% and preferably about 45% to about 70% by weight

to the gum base.

A variety of traditional ingredients such as plasticizers or softeners such as lanolin, stearic acid, sodium
5 stearate, potassium stearate, glyceryl triacetate, glycerin and the like, for example, natural waxes, petroleum waxes, such as polyurethane waxes, paraffin waxes and microcrystalline waxes may also be incorporated into the gum base to obtain a variety of desirable
10 textures and consistency properties. In accordance with the invention, however, these ingredients may be reduced in amount or in some cases, may be eliminated entirely. When present, these individual additional materials are generally employed in amounts of up to about 15% by
15 weight and preferably in amounts of from about 3% to about 10% by weight of the final gum base composition.

The chewing gum composition may additionally include the conventional additives of coloring agents such as
20 titanium dioxide; emulsifiers such as lecithin and glyceryl monostearate; additional fillers such as aluminum hydroxide, alumina, aluminum silicates, calcium carbonate, and talc and combinations thereof; and additional flavoring agents. These fillers may also be
25 used in the gum base in various amounts. Preferably, the amount of fillers when used will vary from about 4% to about 35% by weight of the final chewing gum.

In the instance where sweeteners are utilized in addition
30 to those that may be included in the delivery system, the present invention contemplates the inclusion of those sweeteners well known in the art, including both natural and artificial sweeteners. Thus, additional sweeteners may be chosen from the following non-limiting list:
35 sugars such as sucrose, glucose (corn syrup), dextrose, invert sugar, fructose, polydextrose, cellulose, fibers,

and mixtures thereof, saccharine and its various salts such as the sodium or calcium salt; cyclamic acid and its various salts such as the sodium salt; the dipeptide sweeteners such as aspartame; dihydrochalcone compounds, glycyrrhizin; Stevia Rebaudiana (Stevioside); chloro derivatives of sucrose; dihydroflavinol; hydroxyguaiacol esters; L-amino dicarboxylic acid gem-diamines; L-aminodicarboxylic acid aminoalkenoic acid ester amides; and sugar alcohols such as sorbitol, sorbitol syrup, mannitol, xylitol, and the like. Also contemplated as an additional sweetener is the nonfermentable sugar substitute (hydrogenated starch hydrolysate) which is described in U.S. Reissue Patent No. 26,959. Also contemplated is the synthetic sweetener 3,6-dihydro-6-methyl-1-1,2,3-oxathiazin-4-one-2,2-dioxide, particularly the potassium (acesulfame-K), sodium and calcium salts thereof as described in German Patent No. 2,001,017.7.

Suitable auxiliary flavorings including both natural and artificial flavors, and mints such as peppermint, menthol, artificial vanilla, cinnamon, various fruit flavors, both individual and mixed, and the like are contemplated. The flavorings are generally utilized in amounts that will vary depending upon the amount of the flavor delivery system employed and may, for example, range in amounts of up to about 2% by weight of the final chewing gum composition weight. Thus flavorings may be presented in the delivery system, in the chewing gum composition itself, or both.

The colorants useful in the present invention include the pigments such as titanium dioxide, that may be incorporated in amounts of up to about 1% by weight, and preferably up to about 6% by weight. Also, the colorants may include other dyes suitable for food, drug and cosmetic applications, and known as FD&C dyes and the

like. The materials acceptable for the foregoing spectrum of use are preferably water-soluble.

Illustrative examples include indigoid dye, known as FD&C Blue No. 2, which is the disodium salt of 5,5'-

5 indigotindisulfonic acid. Similarly, the dye known as FD&C Green No. 1 comprises a triphenylmethane dye and is the monosodium salts of 4-[4-N-ethyl-p-sulfobenzylamino)diphenylmethylene]-[1-(N-ethyl-N-p-sulfoniumbenzyl)-2-5-cyclohexadieneimine]. A full
10 recitation of all FD&C and D&C and their corresponding chemical structures may be found in the Kirk-Othmer Encyclopedia of Chemical Technology, in Volume 5, pages 857-884, which text is accordingly incorporated herein by reference.

15

The chewing gums of the invention may be in any form known in the art, such as stick gum, slab gum, chunk gum, shredded gum, hard-coated gum, tableted gum, as well as center-filled gum.

20

A representative process for preparing a chewing gum composition including the inventive flavor delivery system is as follows. The gum base is melted (about 85° to about 90°C), cooled to from 75° to 80°C and placed in
25 a pre-warmed (60°C) standard mixing kettle equipped with sigma blades. Any additional emulsifier for the gum base is then added and mixed in. Next, a portion of the sorbitol and any glycerin is added and mixed in for an additional 3 to 6 minutes. The mixing kettle is cooled
30 and mannitol, and the remainder of the sorbitol and glycerin are then added and mixing is continued. At the time, the unflavored chewing gum temperature is about 39° to about 50°C. The flavor delivery system is then added and incorporated into the base and mixing is continued.
35 Finally, the sweetener material is added and mixed for an additional 1 to 10 minutes. The final gum temperature is

about 39°C-50°C. The chewing gum composition is then discharged from the kettle, rolled, scored and formed into chewing gum pieces.

5 Accordingly, a further embodiment of the present invention relates to a method of preparing a chewing gum composition having improved sustained flavor intensity and release properties which comprises:

10 (1) Preparing a flavor delivery system wherein the flavor is a solution mixture, by the process comprising:

(a) melting a resin component selected from rosins, rosin esters, and mixtures;

(b) adding a polyalkylene wax component to the
15 resin melt, and heating and mixing to form a homogeneous mixture;

(c) adding the flavor component to the melted resin-wax mixture under agitation, and stirring the resulting mixture with external heat withdrawn;

20 (d) the flavor component, resin component and wax component present in amounts by weight of the final delivery system of from 1.0% to 90% for the flavor component, 0.5% to 80% for the resin component and 0.5% to 90% for the wax component;

25 (e) continuing to stir the mixture of Step (d) until it cools to about room temperature; and

(f) recovering the final delivery system.

(2) Adding the resultant delivery system to a
30 homogeneous mixture of a gum base and remaining chewing gum ingredients; and

(3) Forming the resultant mixture into suitable chewing gum shapes.

35 More particularly, the flavor delivery system of the

- present invention may be prepared by a method comprising heating a quantity of the resin component to its melting point and preferably to a temperature of about 90° to about 95°C, after which the wax component may be added
- 5 with continued mixing. The temperature may be raised to 100° - 110°C and mixing is continued until a homogeneous mixture is formed. The temperature of the resulting mixture may then be dropped to a temperature ranging from about 84° to about 86°C. At this point, further heat is
- 10 withdrawn and the addition of the flavor component commences under agitation. For example one-fourth of the flavor component may be added while the resin-wax mixture is maintained under an agitation of 2500 rpm and a temperature ranging from about 84° to about 86°C.
- 15 Thereafter, the rest of the flavor is slowly added under agitation until flavor addition is complete, at which point the temperature of the melt will drop to about 50° to 55°C. During agitation, the container lid of the mixing kettle or vat is kept closed to minimize flavor
- 20 evaporation. The resulting melt is further agitated until mixing is complete, such agitation being maintained, for example, for a period of 3-4 minutes. At this point, the final delivery system is completely prepared.
- 25 In the instance where the emulsifier is added to the flavor delivery system, it is added to the resin-wax mixture while the mixture is maintained at a temperature of 90°C. The remainder of the processing is unchanged.
- 30 In the instance where additional artificial sweetener such as aspartame is included in the preparation of the flavor delivery system, it may be added generally concurrently or sequentially with that of the flavor component.
- 35 As mentioned earlier, the present flavor delivery system

may be readily incorporated into a variety of products including comestible products such as chewing gums including both sugared and sugarless gums, pharmaceutical preparations including tablet coatings, dentifrices and dental compositions and industrial non-edible applications such as aroma packaging and "scratch-and-sniff" applications useful for advertising and novelty flavor and aroma delivery.

The following examples serve to provide further appreciation of the invention but are not meant in any way to restrict the effective scope of the invention. All percentages throughout the specification are by weight percent of the final delivery system unless otherwise indicated.

EXAMPLE 1

In this example, representative formulations of the flavor delivery system of the present system were prepared. The specific components of the flavor delivery system comprised a peppermint flavor oil, the glycerol ester of partially hydrogenated wood gum rosin (Resin Ester #5) and the polyethylene wax known as POLYWAX® AC-6. Twelve flavor formulations were prepared and subsequently incorporated into chewing gum compositions. The systems were prepared in accordance with the method described hereinabove in the individual proportions as set forth in Table 1, below.

TABLE 1
FORMULATION FOR FLAVOR DELIVERY SYSTEM

| <u>INGREDIENTS</u> | <u>RANGES</u> (wt.%) | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> | <u>EX.</u> |
|--------------------|-------------------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|
| | | <u>1</u> | <u>2</u> | <u>3</u> | <u>4</u> | <u>5</u> | <u>6</u> | <u>7</u> | <u>8</u> | <u>9</u> | <u>10</u> | <u>11</u> | <u>12</u> |
| Polyethylene | 0.5-90 | - | 5.0 | 7.5 | 10 | 15 | 25 | 50 | 40 | 30 | 50 | 35 | 20 |
| Resin | 0.5-80 | 55 | 50 | 47.5 | 45 | 40 | 30 | 20 | 30 | 40 | 20 | 25 | 30 |
| 10 Emulsifier | 0-25 | 5 | 5 | 5.0 | 5 | 5 | 5 | 5 | 10 | 0 | 0 | 5 | 10 |
| Liquid Flavor | 1.0-90 | 40 | 40 | 40 | 40 | 40 | 40 | 25 | 20 | 30 | 30 | 35 | 40 |

EXAMPLE 2

In this example, two representative gum compositions were prepared for comparative sensory evaluation testing. The compositions comprised a standard peppermint sugared gum composition which was prepared in accordance with standard gum formulation procedures for this particular gum product. The control sample contained a combination of conventional spray dried and liquid peppermint flavor, while the inventive sample contained, respectively, the flavor delivery system and a combination of liquid flavor and spray dried flavor. The particular percentages of ingredients are set forth in Table 2, below.

TABLE 2

| <u>GUM FORMULA - SUGARED GUM</u> | | |
|----------------------------------|----------------|------------------|
| | <u>CONTROL</u> | <u>INVENTIVE</u> |
| GUM BASE | 21.00 | 21.00 |
| 20 Softeners | 0.95 | 0.95 |
| Carbohydrates | 72.7838 | 71.3088 |
| SWEETENERS - Free and | | |
| encapsulated (3600 ppm) | 3.9162 | 3.9162 |
| 25 FLAVORS | | |
| Spray Dried Flavors | 0.5000 | 0.5000 |
| Liquid Flavor | 0.8500 | 0.2500 |
| Flavor Emulsion | - | 2.1250 |

30 Comparative chew panel testing was conducted with the gum compositions listed in Table 2. The test samples of gum were given to an expert chew panel for evaluation and were rated primarily according to flavor intensity and
35 longevity at intervals of 5, 10, 20 and 30 minutes. The results are set forth in Figure 1.

As can be seen from Figure 1, the Inventive sample scored better than the Control sample and offered both higher and longer lasting flavor delivery. In addition, the Inventive sample provided a uniform release of flavor during the chew, as well as improved flavor perception and reduced bitterness.

EXAMPLE 3

Additional gum samples were prepared to compare the attributes of the present flavor delivery system in anhydrous gum formulations. Accordingly, two samples comprising a control and an inventive sample were formulated in accordance with standard formulation techniques for low moisture gums and possessed the ingredients and amounts as set forth in Table 3, below. Flavor combinations included liquid flavor, spray dried flavor and/or the present flavor delivery system.

TABLE 3

| <u>GUM FORMULA - SUGARLESS</u> | | | |
|--------------------------------|----------------|------------------|--|
| | <u>CONTROL</u> | <u>INVENTIVE</u> | |
| GUM BASE | 23.0 | 24.0 | |
| 25 Softeners | 9.50 | 9.50 | |
| Carbohydrates | 61.6665 | 59.2665 | |
| SWEETENERS - Free and | | | |
| encapsulated (4400 ppm) | 4.4335 | 4.4335 | |
| 30 FLAVORS | | | |
| Spray Dried Flavors | 0.2000 | 0.3000 | |
| Liquid Flavor | 1.2000 | 0.9000 | |
| Flavor Emulsion | - | 1.6000 | |

35

Comparative chew panel testing was then conducted with

the samples set forth in Table 3. As with Example 2, an expert chew panel was asked to evaluate the samples for the same attributes, and to offer evaluations at the same intervals. The ratings of the samples by the panel
5 members are set forth in Figure 2.

As can be seen from Figure 2, the Inventive sample offered a greater flavor intensity which continued throughout the test period. The Inventive sample also
10 exhibited substantially greater flavor extension and provided more flavor release even after twenty minutes.

This invention may be embodied in other forms or carried out in other ways without departing from the spirit or
15 essential characteristics thereof. The present disclosure is therefore to be considered as in all respects illustrative and not restrictive, the scope of the invention being indicated by the appended Claims, and all changes which come within the meaning and range of
20 equivalency are intended to be embraced therein.

WHAT IS CLAIMED IS:

- 1 1. A flavor delivery system offering improved
2 uniformity, extension and delay of flavor release and
3 temperature stability and reduced bitterness, comprising
4 a non-aqueous solution mixture of:
5 (a) a flavor component in an amount of from about
6 1.0% to about 90% by weight of the final delivery system;
7 (b) a resin component, said resin component
8 comprising a rosin, in an amount of from about 0.5% to
9 about 80% by weight of the final delivery system; and
10 (c) a polyalkylene wax in an amount of from about
11 0.5% to about 90% by weight of the final delivery system;
12 wherein said resin component and said polyalkylene
13 wax component protect, hold and mask the aroma of said
14 flavor component even at high temperatures, whereby said
15 flavor delivery system may be formulated with higher
16 concentrations of said flavor component and exhibits said
17 reduced bitterness and said combined delayed and extended
18 flavor release.
- 1 2. The flavor delivery system of Claim 1 wherein said
2 flavor component comprises a flavor oil.
- 1 3. The flavor delivery system of Claim 2 wherein said
2 flavor oil is selected from the group consisting of
3 spearmint oil, cinnamon oil, oil of wintergreen
4 (methylsalicylate), peppermint oil, lemon oil, orange
5 oil, grape oil, lime oil, grapefruit oil, apple essence,
6 strawberry essence, cherry essence, pineapple essence,
7 banana oil and mixtures thereof.
- 1 4. The flavor delivery system of Claim 1 wherein the
2 flavor component is present in an amount of about 35% to
3 about 65% by weight, the resin is present in an amount of
4 about 20% to about 55% by weight, and the polyalkylene

5 wax is present in an amount of about 5.0% to about 50% by
6 weight.

1 5. The flavor delivery system of Claim 1 wherein the
2 flavor component is present in an amount of about 30% to
3 about 45% by weight, the resin is present in an amount of
4 about 35% to about 55% by weight, and the polyalkylene
5 wax is present in an amount of about 2.5% to about 15% by
6 weight.

1 6. The flavor delivery system of Claim 1 wherein the
2 polyalkylene wax comprises a polyethylene wax having a
3 molecular weight ranging from about 500 to about 10,000.

1 7. The flavor delivery system of Claim 1 wherein the
2 resin component is selected from the group consisting of
3 rosins, rosin esters, and mixtures.

1 8. The flavor delivery system of Claim 7 wherein the
2 resin component is selected from the group consisting of
3 pentaerythritol ester of partially hydrogenated wood
4 rosin, pentaerythritol ester of wood rosin, glycerol
5 ester of wood rosin, glycerol ester of partially
6 dimerized rosin, glycerol ester of polymerized rosin,
7 glycerol ester of tall oil rosin, glycerol ester of wood
8 rosin and partially hydrogenated wood rosin and partially
9 hydrogenated methyl ester of rosin, such as polymers of
10 alpha-pinene or beta-pinene; terpene resins including
11 polyterpene; and mixtures thereof.

1 9. The flavor delivery system of Claim 1 further
2 including an emulsifier.

1 10. The flavor delivery system of Claim 9 wherein said
2 emulsifier is present in an amount of up to about 25% by
3 weight of the final delivery system.

1 11. The flavor delivery system of Claim 9 wherein said
2 emulsifier is present in an amount of from about 10% to
3 about 20% by weight of the final delivery system.

1 12. The flavor delivery system of Claim 9 wherein said
2 emulsifier is present in an amount of from about 5% to
3 about 10% by weight of the final delivery system.

1 13. The flavor delivery system of Claim 9 wherein the
2 emulsifier is selected from the group consisting of
3 monoglycerides, diglycerides and triglycerides of fatty
4 acids, polyglycerol esters, and mixtures thereof.

1 14. The flavor delivery system of Claim 9 wherein the
2 emulsifier is selected from the group consisting of
3 lecithin, stearates, ester derivatives of stearates,
4 palmitates, ester derivatives of palmitates, oleates,
5 ester derivatives of oleates, glycerides, sucrose
6 polyesters, polyglycerol esters and mixtures thereof.

1 15. The flavor delivery system of Claim 6 wherein a
2 mixture of polyethylene waxes of differing molecular
3 weight is present.

1 16. The flavor delivery system of Claim 1 further
2 including up to 25% by weight of a sweetener.

1 17. The flavor delivery system of Claim 16 wherein the
2 sweetener is a natural or artificial high intensity
3 sweetener selected from the group consisting of amino
4 acid-based sweeteners, dipeptide sweeteners,
5 glycyrrhizin, saccharin and its salts, acesulfame salts,
6 cyclamates, steviosides, talin, sucralose,
7 dihydrochalcone compounds and mixtures thereof.

1 18. The flavor delivery system of Claim 16 wherein the

2 sweetener is acesulfame-K.

1 19. The flavor delivery system of Claim 1 in solid
2 particulate form.

1 20. A chewing gum composition offering improved flavor
2 intensity and longevity, comprising a gum base, a
3 sweetener and a flavor delivery system, said flavor
4 delivery system offering improved flavor protection and a
5 combination of delayed and extended release
6 characteristics in conjunction with reduced bitterness,
7 said flavor delivery system comprising a non-aqueous
8 solution mixture of:

9 (a) a flavor component in an amount of from about
10 1.0% to about 90% by weight of the final delivery system;

11 (b) a resin component, said resin component
12 comprising a rosin, in an amount of from about 0.5% to
13 about 80% by weight of the final delivery system; and

14 (c) a polyalkylene wax in an amount of from about
15 0.5% to about 90% by weight of the final delivery system;

16 wherein said resin component and said polyalkylene
17 wax component protect, hold and mask the aroma of said
18 flavor component even at high temperatures, whereby said
19 flavor delivery system may be formulated with higher
20 concentrations of said flavor component and exhibits said
21 reduced bitterness and said combined delayed and extended
22 flavor release.

1 21. The chewing gum composition of Claim 20, wherein the
2 gum base comprises an elastomer selected from the group
3 consisting of natural rubber, synthetic rubber and
4 mixtures thereof.

1 22. The chewing gum composition of Claim 21, wherein the
2 gum base elastomer is selected from the group consisting
3 of chicle, jelutong, balata, gutta-percha, lechi-capsi,

4 sorva, butadiene-styrene copolymers, polyisobutylene,
5 isobutylene-isoprene copolymers, polyethylene,
6 polyvinylacetate, and mixtures thereof.

1 23. The chewing gum composition of Claim 22, wherein the
2 gum base is present in amounts of about 5% to about 85%
3 by weight of the final chewing gum composition.

1 24. The chewing gum composition of Claim 20 further
2 including fillers, coloring agents, flavoring agents,
3 softeners, plasticizers, elastomers, elastomer solvents,
4 sweetening agents and mixtures thereof.

1 25. A method of preparing a chewing gum composition
2 having improved sustained flavor intensity and release
3 properties which comprises:

4 (1) Preparing a flavor delivery system wherein the
5 flavor is a solution mixture, by the process comprising

6 (a) melting a resin component selected from
7 rosins, rosin esters, and mixtures;

8 (b) adding a polyalkylene wax component to the
9 resin melt, and heating and mixing to form a homogeneous
10 mixture;

11 (c) adding the flavor component to the melted
12 resin-wax mixture under agitation, and stirring the
13 resulting mixture with external heat withdrawn;

14 (d) the flavor component, resin component and
15 wax component present in amounts by weight of the final
16 delivery system of from 1.0% to 90% for the flavor
17 component 0.5% to 80% for the resin component and 0.5% to
18 90% for the wax component;

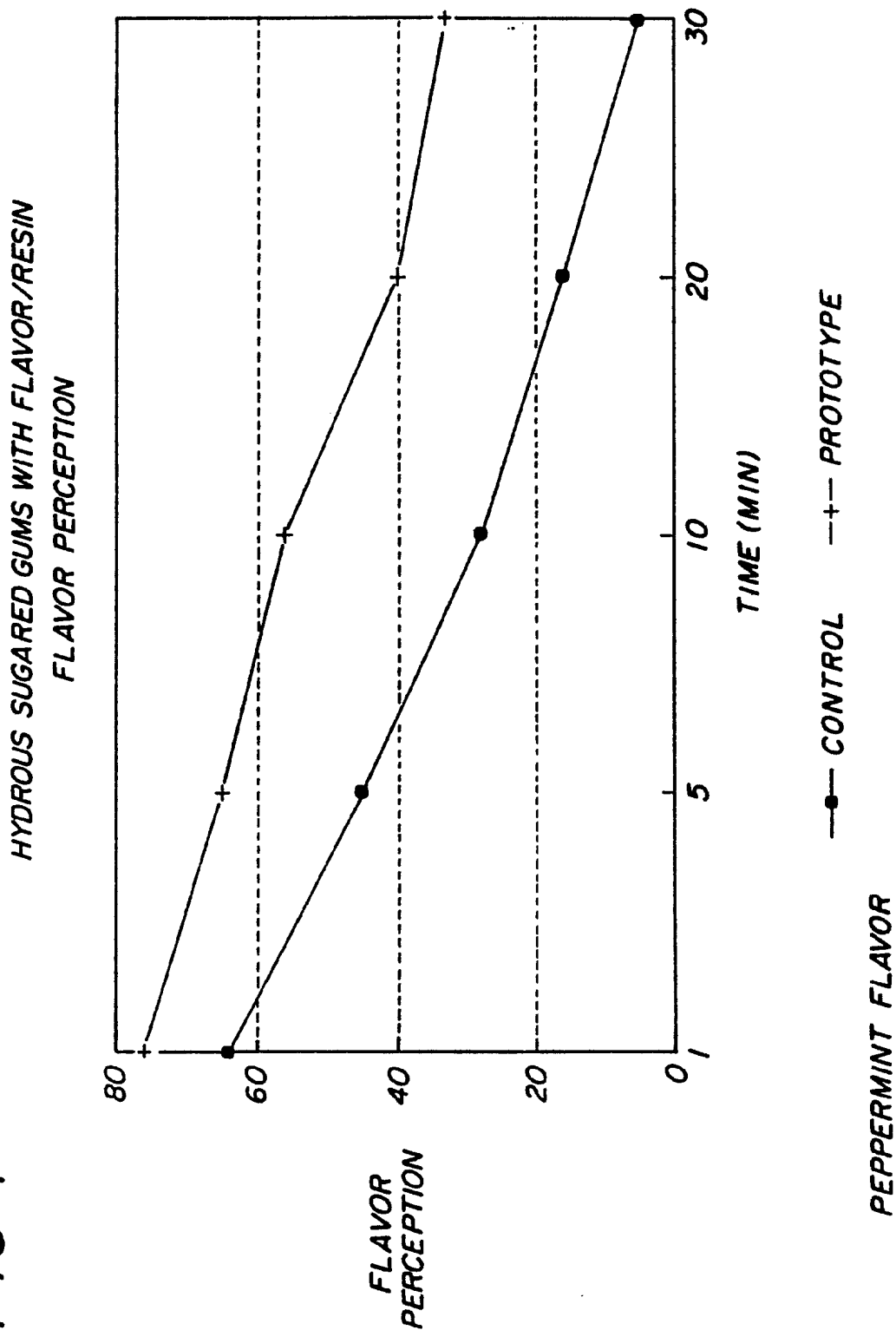
19 (e) continuing to stir the mixture of Step (d)
20 until it cools to about room temperature; and

21 (f) recovering the final delivery system.

22 (2) Adding the resultant delivery system to a
23 homogeneous mixture of a gum base and remaining chewing

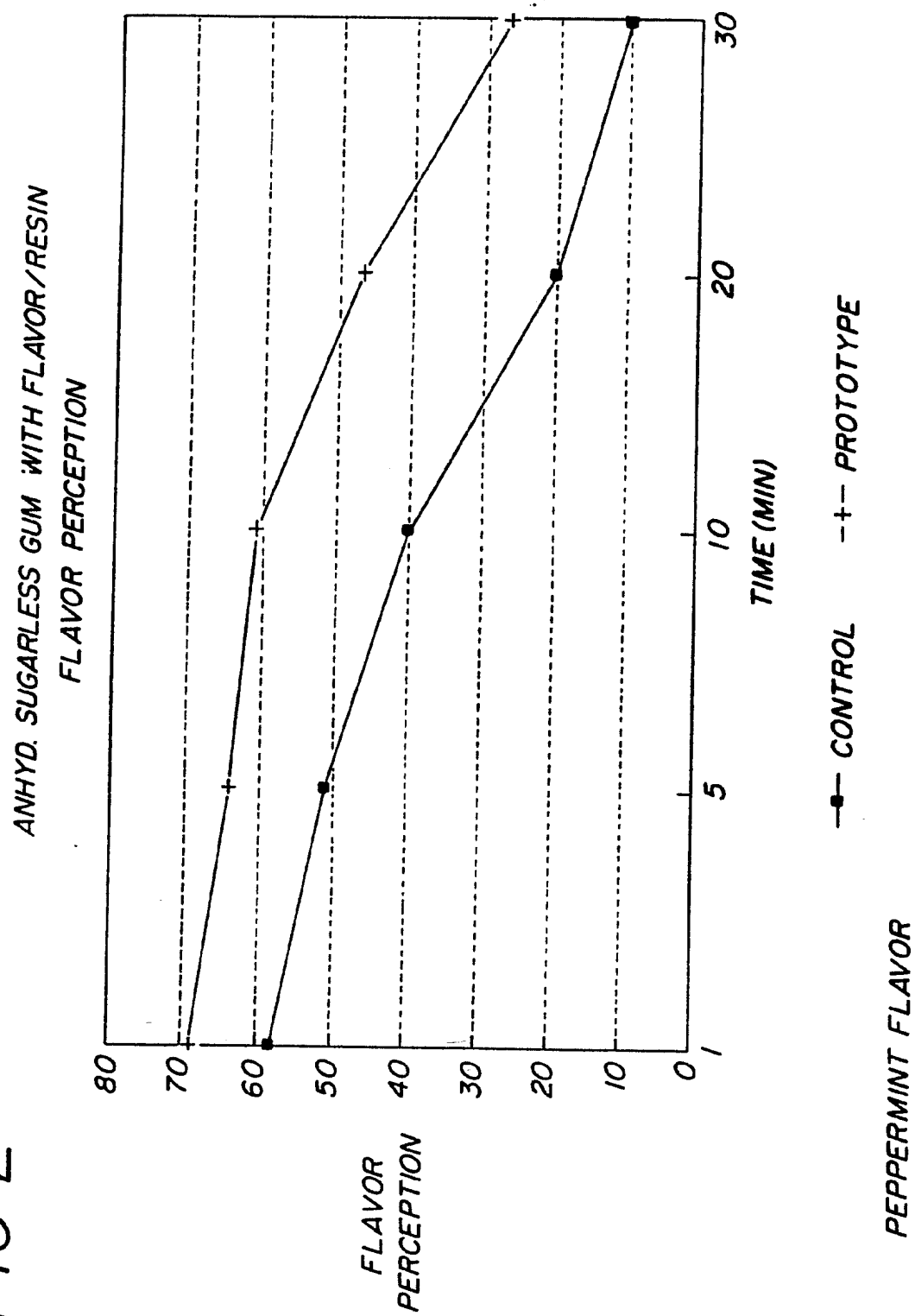
- 24 gum ingredients; and
25 (3) Forming the resultant mixture into suitable
26 chewing gum shapes.
- 1 26. The flavor delivery system of Claim 1 incorporated
2 into a confectionery composition.
- 1 27. The flavor delivery system of Claim 1 incorporated
2 into a pharmaceutical composition.
- 1 28. The flavor delivery system of Claim 1 incorporated
2 into a food product.
- 1 29. The flavor delivery system of Claim 1 incorporated
2 into a dentifrice composition or denture adhesive.
- 1 30. The flavor delivery system of Claim 1 incorporated
2 into a coating for scratch-and-sniff products and aroma
3 packaging products.

FIG-1



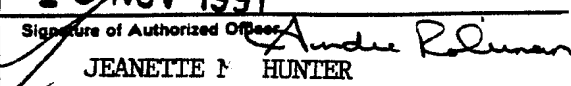
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FIG-2



INTERNATIONAL SEARCH REPORT

International Application No. PCT/US91/05348

| | | |
|--|--|-------------------------------------|
| I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶ | | |
| According to International Patent Classification (IPC) or to both National Classification and IPC IPC(5): A23 G 3/30; A23L 1/058; A23L 1/22; A23L 1/226 U.S. C1: 426/3,4,5,6,650,651; 424/48,49,440; 252/315.1, 315.2; 512/4,9,65,944 | | |
| II. FIELDS SEARCHED | | |
| Minimum Documentation Searched ⁷ | | |
| Classification System | Classification Symbols | |
| U.S. | 426/3,4,5,6,650, 651; 424/48,49,440; 252/315.1, 315.2; 512/4,9,65,944 | |
| Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸ | | |
| | | |
| III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹ | | |
| Category ¹⁰ | Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹² | Relevant to Claim No. ¹³ |
| A | U.S., A, 4,971,785 WILSON ET. AL. 20 NOVEMBER 1990 | 1-30 |
| A | U.S., A, 4,528,125 ALDERMAN ET. AL. 09 JULY 1985 | 1-30 |
| A | U.S., A, 4,590,075 WEI ET. AL. 20 MAY 1986 | 1-30 |
| A | U.S., A, 4,906,480 KASHET 06 MARCH 1990 | 1-30 |
| A | U.S., A, 4,908,212 KWON ET. AL. 13 MARCH 1990 | 1-30 |
| A | U.S., A, 4,752,481 DOKUZOVIC 21 JUNE 1988 | 1-30 |
| <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>¹⁰ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p> </div> </div> | | |
| IV. CERTIFICATION | | |
| Date of the Actual Completion of the International Search | Date of Mailing of this International Search Report | |
| 05 NOVEMBER 1991 (05-11-91) | 19 NOV 1991 | |
| International Searching Authority | Signature of Authorized Officer | |
| ISA/US |  JEANETTE F. HUNTER | |

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

| | | |
|---|---|------|
| A | U.S., A, 4,803,082 CHERUKURI ET. AL 07 FEBRUARY 1989 | 1-30 |
| A | U.S., A, 4,568,560 SCHOBEL 04 FEBRUARY 1986 | 1-30 |
| A | U.S., A, 4,508,744 KRUGER JR. ET. AL 02 APRIL 1985 | 1-30 |

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE ¹

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers _____, because they relate to subject matter ¹² not required to be searched by this Authority, namely:

2. ☐ Claim numbers _____, because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out ¹³, specifically:

3. ☐ Claim numbers _____, because they are dependent claims not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING ²

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.
2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

- ☐ The additional search fees were accompanied by applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

| Category * | Citation of Document, with indication, where appropriate, of the relevant passages | Relevant to Claim No |
|------------|--|----------------------|
| A | U.S., A, 4,485,118 CARROL ET. AL 27 NOVEMBER 1984 | 1-30 |